

**ATTACHMENT B3**

**QUALITY ASSURANCE OBJECTIVES AND DATA VALIDATION  
TECHNIQUES FOR WASTE CHARACTERIZATION SAMPLING AND  
ANALYTICAL METHODS**

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## ATTACHMENT B3

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### QUALITY ASSURANCE OBJECTIVES AND DATA VALIDATION TECHNIQUES FOR WASTE CHARACTERIZATION SAMPLING AND ANALYTICAL METHODS

#### B3-1 Validation Methods

The Permittees shall require the generator/storage sites (**sites**) to perform validation of all data (qualitative as well as quantitative) so that data used for Waste Isolation Pilot Plant (**WIPP**) compliance programs will be of known and acceptable quality. Validation includes a quantitative determination of precision, accuracy, completeness, and method detection limits (as appropriate) for analytical data (headspace Volatile Organic Compounds (**VOC**), total VOCs, Semivolatile Organic Compounds (**SVOC**), and metals data). Quantitative data validations shall be performed according to the conventional methods outlined below (equations B3-1 through B3-8). These quantitative determinations will be compared to the Quality Assurance Objectives (**QAOs**) specified in Sections B3-2 through B3-9. A qualitative determination of comparability and representativeness will also be performed.

The qualitative data or descriptive information generated by radiography and visual examination is not amenable to statistical data quality analysis. However, radiography and visual examination are complementary techniques yielding similar data for determining the waste matrix code. The waste matrix code is determined to ensure that the container is properly included in the appropriate waste stream.

Data validation will be used to assess the quality of waste characterization data collected based upon project precision, accuracy, completeness, comparability, and representativeness objectives. These objectives are described below:

#### Precision

Precision is a measure of the mutual agreement among multiple measurements of a single analyte, either by the same method or by different methods. Precision is either expressed as the relative percent difference (**RPD**) for duplicate measurements or as the percent relative standard deviation (**%RSD**) for three or more replicate measurements. For duplicate measurements, the precision expressed as the RPD is calculated as follows:

$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} \times 100 \quad (B3-1)$$

where  $C_1$  and  $C_2$  are the two values obtained by analyzing the duplicate samples.  $C_1$  is the larger of the two observed values.

For three or more replicate measurements, the precision expressed as the %RSD is calculated as follows:

$$\%RSD = \frac{s}{y_{mean}} \times 100 \quad (B3-2)$$

where  $s$  is the standard deviation and  $y_{mean}$  is the mean of the replicate sample analyses.

The standard deviation,  $s$ , is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^n (y_i - y_{mean})^2}{n - 1}} \quad (B3-3)$$

where  $y_i$  is the measured value of the  $i$ th replicate sample analysis measurement, and  $n$  equals the number of replicate analyses.

Another aspect of precision is associated with analytical equipment calibration. In these instances, the percent difference (**%D**) between multiple measurements of an equipment calibration standard shall be calculated as follows:

$$\%D = \frac{|C_1 - C_2|}{C_1} \times 100 \quad (B3-4)$$

where  $C_1$  is the initial measurement and  $C_2$  is the second or other additional measurement.

### Accuracy

Accuracy is the degree of agreement between a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) and the true or known concentration. Accuracy is determined as the percent recovery (**%R**).

For situations where a standard reference material is used, the **%R** is calculated as follows:

$$\%R = \frac{C_m}{C_{sm}} \times 100 \quad (B3-5)$$

where  $C_m$  is the measured concentration value obtained by analyzing the sample and  $C_{sm}$  is the "true" or certified concentration of the analyte in the sample.

For measurements where matrix spikes are used, the **%R** is calculated as follows:

$$\%R = \frac{S - U}{C_{sc}} \times 100 \quad (B3-6)$$

where S is the measured concentration in the spiked aliquot, U is the measured concentration in the unspiked aliquot, and  $C_{sc}$  is the actual concentration of the spike added.

#### Method Detection Limit

The method detection limit (**MDL**) is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The MDL for all quantitative measurements (except for those using Fourier Transform Infrared Spectroscopy [**FTIRS**]) is defined as follows:

$$MDL = t_{(n-1, 1-\alpha=.99)} \times s \quad (B3-7)$$

where  $t_{(n-1, 1-\alpha=.99)}$  is the t-distribution value corresponding to a 99 percent confidence level with n-1 degrees of freedom, n is the number of observations, and s is the standard deviation of replicate measurements.

For headspace-gas analysis using FTIRS, MDL is defined as follows:

$$MDL = 3s \quad (B3-8)$$

where s is the standard deviation. Initially, a minimum of seven samples spiked at a level of three to five times the estimated MDL and analyzed on non-consecutive days must be used to establish the MDLs. MDLs should be updated using the results of the laboratory control sample or on-line control samples.

#### Completeness

Completeness is a measure of the amount of valid data obtained from the overall measurement system compared to the amount of data collected and submitted for analysis. Completeness must be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Completeness, expressed as the percent complete (**%C**), is calculated as follows:

$$\%C = \frac{V}{n} \times 100 \quad (B3-9)$$

where V is the number of valid sampling or analytical results obtained and n is the number of samples submitted for analysis.

## Comparability

Comparability is the degree to which one data set can be compared to another. Comparability of data generated at different sites will be ensured through the use of standardized, approved testing, sampling, preservation, and analytical techniques and by meeting the QAOs specified in Sections B3-2 through B3-9.

The comparability of waste characterization data shall be ensured through the use of generator/storage site data usability criteria. The Permittees shall ensure that data usability criteria are consistently established and used by the generator/storage sites to assess the usability of analytical and testing data. The criteria shall address, as appropriate, the following:

- Definition or reference of criteria used to define and assign data qualifier flags based on Quality Assurance Objective results,
- Criteria for assessing the useability of data impacted by matrix interferences,
- Criteria for assessing the useability of data based upon positive and negative bias as indicated by quality control data, of data qualifiers, and qualifier flags,
- Criteria for assessing the useability of data due to
  - Severe matrix effects,
  - Misidentification of compounds,
  - Gross exceedance of holding times,
  - Failure to meet calibration or tune criteria
- Criteria for assessing the useability of data that does not meet minimum detection limit requirements.

The Permittees shall be responsible for evaluating generator/storage site data useability and shall assess implementation through the generator/storage site audit.

## Representativeness

Representativeness is the degree to which sample data represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that concerns the proper design of the sampling program.

Representativeness of waste containers from waste streams subjected to headspace gas, homogeneous solids, and soil/gravel sampling and analysis will be validated, through documentation, that a true random sample with an adequate population was identified and collected consistent with Permit Attachment B2, Section B2-1. Since representativeness is a quality characteristic that expresses the degree to which a sample or group of samples represents the population being studied, the random selection of waste containers ensures representativeness on a Program level. The Permittees shall require the Site Project Manager to document that the selected waste containers from within a waste stream were randomly selected. Sampling personnel shall verify that proper procedures are followed to ensure that

1 samples are representative of the waste contained in a particular waste container or a waste  
2 stream.

### 3 Identification of Tentatively Identified Compounds

4 In accordance with SW-846 convention, identification of compounds detected by gas  
5 chromatography/mass spectrometry methods that are not on the list of target analytes shall be  
6 reported. Both composited and individual container headspace gas, volatile analysis  
7 (TCLP/Totals), and semi-volatile (TCLP/Totals) shall be subject to tentatively identified  
8 compound (**TIC**) reporting. These TICs for GC/MS Methods are identified in accordance with the  
9 following SW-846 criteria:

- 10 ● Relative intensities of major ions in the reference spectrum (ions greater than  
11 10% of the most abundant ion) should be present in the sample spectrum.
- 12 ● The relative intensities of the major ions should agree within  $\pm 20$  percent.
- 13 ● Molecular ions present in the reference spectrum should be present in the  
14 sample spectrum.
- 15 ● Ions present in the sample spectrum but not in the reference spectrum should be  
16 reviewed for possible background contamination or presence of coeluting  
17 compounds.
- 18 ● Ions present in the reference spectrum but not in the sample spectrum should be  
19 reviewed for possible subtraction from the sample spectrum because of  
20 background contamination or coeluting peaks.
- 21 ● The reference spectra used for identifying TICs shall include, at minimum, all of  
22 the available spectra for compounds that appear in the 20.4.1.200 NMAC  
23 (incorporating 40 CFR Part 261) Appendix VIII list. The reference spectra may be  
24 limited to VOCs when analyzing headspace gas samples.
- 25 ● TICs for headspace gas analyses that are performed through FTIR analyses  
26 shall be identified in accordance with the specifications of SW-846 Method 8410.

27 TICs shall be reported as part of the analytical batch data reports for GC/MS Methods in  
28 accordance with the following minimum criteria:

- 29 ● a TIC in an individual container headspace gas or solids sample shall be  
30 reported in the analytical batch data report if the TIC meets the SW-846  
31 identification criteria listed above and is present with a minimum of 10%  
32 of the area of the nearest internal standard.
- 33 ● a TIC in a composited headspace gas sample that contains 2 to 5  
34 individual container samples shall be reported in the analytical batch data  
35 report if the TIC meets the SW-846 identification criteria listed above and

is present with a minimum of 2% of the area of the nearest internal standard.

- a TIC in a composited headspace gas sample that contains 6 to 10 individual container samples shall be reported in the analytical batch data report if the TIC meets the SW-846 identification criteria listed above and is present with a minimum of 1% of the area of the nearest internal standard.
- a TIC in a composited headspace gas sample that contains 11 to 20 individual container samples shall be reported in the analytical batch data report if the TIC meets the SW-846 identification criteria listed above and is present with a minimum of 0.5% of the area of the nearest internal standard.

TICs that meet the SW-846 identification criteria, are reported in 25 percent of all waste containers sampled from a given waste stream, and that appear in the 20.4.1.200 NMAC (incorporating 40 CFR §261) Appendix VIII list, will be compared to acceptable knowledge data to determine if the TIC is a listed waste in the waste stream. TICs identified through headspace gas analyses that meet the Appendix VIII list criteria and the 25 percent reporting criteria for a waste stream will be added to the headspace gas waste stream target list regardless of the hazardous waste listing associated with the waste stream. TICs reported from the Totals VOC or SVOC analyses may be excluded from the target analyte list for a waste stream if the TIC is a constituent in an F-listed waste whose presence is attributable to waste packaging materials or radiolytic degradation from acceptable knowledge documentation. If a listed waste constituent TIC cannot be attributed to waste packaging materials, radiolysis, or other origins, the constituent will be added to the target analyte list and new hazardous waste numbers will be assigned, if appropriate. TICs subject to inclusion on the target analyte list that are toxicity characteristic parameters shall be added to the target analyte list regardless of origin because the hazardous waste designation for these numbers is not based on source. However, for toxicity characteristic and non-toxic F003 constituents, the site may take concentration into account when assessing whether to add a hazardous waste number. If a target analyte list for a waste stream is expanded due to the presence of TICs, all subsequent samples collected from that waste stream will be analyzed for constituents on the expanded list.

### B3-2 Headspace-Gas Sampling

#### Quality Assurance Objectives

The precision and accuracy of the container headspace-gas sampling operations must be assessed by analyzing field QC headspace-gas samples. These samples must include equipment blanks, field reference standards, field blanks, and field duplicates. If the QAOs described below are not met, a nonconformance report must be prepared, submitted, and resolved (Section B3-13).

1     Precision

2     The precision of the headspace-gas sampling and analysis operation must be assessed by  
3     sequential collection of field duplicates for manifold sampling operations or simultaneous  
4     collection of field duplicates for direct canister sampling operations for VOCs determination.  
5     Corrective actions must be taken if the RPD exceeds 25 percent for any analyte found greater  
6     than the PRQL in both of the duplicate samples.

7     Accuracy

8     A field reference standard must be collected using headspace-gas sampling equipment to  
9     assess the accuracy of the headspace-gas sampling operation at a frequency of one field  
10    reference standard for every 20 containers sampled or per sampling batch. Corrective action  
11    must be taken if the %R of the field-reference standard is less than 70 or greater than 130.

12   Field blanks must also be collected at a frequency of 1 field blank for every 20 containers or  
13   sampling batch sampled to assess possible contamination in the headspace gas sampling  
14   method. Equipment blanks must also be collected at a frequency of 1 equipment blank for each  
15   equipment cleaning batch to assess possible contamination in the equipment cleaning method.  
16   Corrective actions must be taken if the blank exceeds three times the MDLs listed for any of the  
17   compounds listed in Table B3-2.

18   Completeness

19   Sampling completeness shall be expressed as the number of valid samples collected as a  
20   percent of the total number of samples collected for each waste stream. A valid sample is  
21   defined as a sample collected in accordance with approved sampling methods and the  
22   container was properly prepared for sampling (e.g., the polyliner was vented to the container  
23   headspace). The Permittees shall require participating sampling facilities to achieve a minimum  
24   90 percent completeness. The amount and type of data that may be lost during the headspace-  
25   gas sampling operation cannot be predicted in advance. The Permittees shall require the Site  
26   Project Manager to evaluate the importance of any lost or contaminated headspace-gas  
27   samples and take corrective action as appropriate.

28   Comparability

29   Consistent use and application of uniform procedures and equipment, as specified in Permit  
30   Attachment B1 and application of data useability criteria, should ensure that headspace gas  
31   sampling operations are comparable when sampling headspace at the different sampling  
32   facilities. The Permittees shall require each site to take corrective actions if uniform procedures,  
33   equipment, or operations are not followed without approved and justified deviations. In addition,  
34   laboratories analyzing samples must successfully participate in the Performance Demonstration  
35   Program (**PDP**) (DOE, 2003).

36   Representativeness

37   Specific headspace-gas sampling steps to ensure samples are representative include:

- Selection of the correct Drum Age Criteria (**DAC**) Scenario and waste packaging configuration and meeting DAC equilibrium times.
- A sample canister cleaning and leak check after assembly
- Sampling equipment cleaning or disposal after use
- Sampling equipment leak check after sample collection
- Use of sample canisters with passivated internal surfaces
- Use of low-internal-volume sampling equipment
- Collection of samples with a low-sample volume to available headspace volume ratio (less than 10 percent of the headspace when the headspace can be determined)
- Careful and documented pressure regulation of all activities specified in Attachment B1, Section B1-1
- Performance audits
- Collection of equipment blanks, field reference standard, field blanks, and field duplicates at the specified frequencies.
- Manifold pressure sensors and temperature sensors calibrated before initial use and annually using NIST, or equivalent standards.
- OVA calibrated daily, prior to first use, or as necessary according to manufacturers specifications.

Failure to perform the checks at the prescribed frequencies would result in corrective actions.

### B3-3 Sampling of Homogeneous Solids and Soils/Gravel

#### Quality Assurance Objectives

To ensure that sampling is conducted in a representative manner on a waste-stream basis for waste containers containing homogeneous solids and soil/gravel, samples must be collected randomly in both the horizontal and vertical planes of each container's waste. For waste containers that contain homogeneous solids and soil/gravel in smaller containers (e.g., 1 gal [4.0 L] poly bottles) within the waste container, one randomly chosen smaller container must be sampled from each container.

## Precision

Sampling precision must be determined by collecting and sampling field duplicates (e.g., co-located cores or co-located samples as described in Permit Attachment B1-2b(1)) once per sampling batch or once per week during sampling operations, whichever is more frequent. A sampling batch is a suite of homogeneous solids and soil/gravel samples collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the first sample in the batch. The Permittees shall require the Site Project Manager to calculate and report the RPD between co-located core/samples.

The recommended method for establishing acceptance criteria for co-located cores and co-located samples is the F-test method because the F-Test: 1) does not require potentially arbitrary groupings into batches, 2) is based on exact distributions, and 3) is more likely to detect a change in the process. When a sufficient number of samples are collected (25 to 30 pairs of co-located cores or samples), control charts of the RPD will be developed for each constituent and for each waste matrix or waste type (e.g., pyrochemical salts or organic sludges). The limits for the control chart will be three standard deviations above or below the average RPD. Once constructed, RPDs for additional co-located pairs will be compared with the control chart to determine whether or not the co-located cores are acceptable. Periodically, the control charts will be updated using all available data.

The statistical test will involve calculating the variance for co-located cores and samples by pooling the variances computed for each pair of duplicate results. The variance for the waste stream will be computed excluding any data from containers with co-located cores, because the test requires the variance estimates to be independent. All data must be transformed to normality prior to computing variances and performing the test. The test hypothesis is evaluated using the F distribution and the method for testing the difference in variances.

## Accuracy

Sampling accuracy through the use of standard reference materials shall not be measured. Because waste containers containing homogeneous solids and soil/gravel with known quantities of analytes are not available, sampling accuracy cannot be determined. However, sampling methods and requirements described are designed to minimize sample degradation and hence maximize sampling accuracy.

Sampling accuracy as a function of sampling cross-contamination will be measured. Equipment blanks will be collected at a frequency of once per equipment cleaning batch. Corrective actions must be taken if the blank exceeds three times the MDLs (PRDLs for metals) listed for any of the compounds or analytes listed in Tables B3-4, B3-6, and B3-8. Equipment blanks will be collected from the following equipment types:

- Fully assembled coring tools
- Liners cleaned separately from coring tools
- Miscellaneous sampling equipment that is reused (bowls, spoons, chisels)

## Completeness

Sampling completeness shall be expressed as the number of valid samples collected as a percent of the total number of samples collected for each waste stream. A valid sample is any sample that is collected from a randomly selected container using randomly selected horizontal and vertical planes in accordance with approved sampling methods. The Permittees shall require participating sampling facilities to achieve a minimum 90 percent completeness.

## Comparability

Consistent use and application of uniform procedures, sampling equipment, and measurement units must ensure that sampling operations are comparable. Consistent application of data useability criteria will also ensure comparability. In addition, the Permittees shall require laboratories analyzing samples to successfully participate in the PDP (DOE, 2005).

## Representativeness

Specific steps to ensure the representativeness of samples include the following for both waste containers and smaller containers:

- Coring tools and sampling equipment must be clean prior to sampling.
- The entire depth of the waste minus a site defined approved safety factor must be cored, and the core collected must have a length greater than or equal to 50 percent of the depth of the waste. This is called the core recovery and is calculated as follows:

$$\text{Core recovery (percent)} = \frac{y}{x} \times 100 \quad (\text{B3-10})$$

where

x = the depth of the waste in the container

y = the length of the core collected from the waste.

- Coring operations and tool selection should be designed to minimize alteration of the in-place waste characteristics. Minimal waste disturbance must be verified by visually examining the core and describing the observation (e.g., undisturbed, cracked, or pulverized) in the field logbook.

If core recovery is less than 50 percent of the depth of the waste, a second coring location shall be randomly selected. The core with the best core recovery shall be used for sample collection.

One randomly selected container within a container will be chosen if the container contains individual waste containers.

## B3-4 Non Destructive Examination Methods

### Quality Assurance Objectives

The QAOs for non destructive examination (**NDE**) are detailed in this section. NDE can be either radiography or visual examination (**VE**). If the QAOs described below are not met, then corrective action shall be taken. It should be noted that NDE does not have a specific MDL because it is primarily a qualitative determination. The objective of NDE for the program is to determine the physical waste form, the absence of prohibited items, and additional waste characterization techniques that may be used based on the Summary Category Groups (i.e., S3000, S4000, S5000). The Permittees shall require each site to describe all activities required to achieve these objectives in the site quality assurance project plan (**QAPjP**) and standard operating procedures (**SOP**).

#### B3-4a Radiography

Data to meet these objectives must be obtained from a video and audio recorded scan provided by trained radiography operators at the sites. Results must also be recorded on a radiography data form. The precision, accuracy, completeness, and comparability objectives for radiography data are presented below.

##### Precision

Precision is maintained by reconciling any discrepancies between two radiography operators with regard to identification of the waste matrix code, liquids in excess of TSDF-WAC limits, and compressed gases through independent replicate scans and independent observations. Additionally, the precision of radiography is verified prior to use by tuning precisely enough to demonstrate compliance with QAOs through viewing an image test pattern.

##### Accuracy

Accuracy is obtained by using a target to tune the image for maximum sharpness and by requiring operators to successfully identify 100 percent of the required items in a training container during their initial qualification and subsequent requalification.

##### Completeness

A video and audio media recording of the radiography examination and a validated radiography data form will be obtained for 100 percent of the waste containers subject to radiography. All video and audio media recordings and radiography data forms will be subject to validation as indicated in Section B3-10.

##### Comparability

The comparability of radiography data from different operators shall be enhanced by using standardized radiography procedures and operator qualifications.

1     B3-4b Visual Examination

2     Results must be recorded on a VE data form. The precision, accuracy, completeness, and  
3     comparability objectives for VE data are presented below.

4     Precision

5     Precision is maintained by reconciling any discrepancies between the operator and the  
6     independent technical reviewer with regard to identification of waste matrix code, liquids in  
7     excess of TSDF-WAC limits, and compressed gases.

8     Accuracy

9     Accuracy is maintained by requiring operators to pass a comprehensive examination and  
10    demonstrate satisfactory performance in the presence of the VE expert during their initial  
11    qualification and subsequent requalification.

12    Completeness

13    A validated VE data form will be obtained for 100 percent of the waste containers subject to VE.

14    Comparability

15    The comparability of VE data from different operators shall be enhanced by using standardized  
16    VE procedures and operator qualifications.

17    B3-5 Gas Volatile Organic Compound Analysis

18    Quality Assurance Objectives

19    The development of data quality objective (**DQOs**) specifically for this program has resulted in  
20    the QAOs listed in Table B3-2. The specified QAOs represent the required quality of data  
21    necessary to draw valid conclusions regarding program objectives. WAP-required limits, such  
22    as the program required quantitation limits (**PRQL**) associated with VOC analysis, are specified  
23    to ensure that the analytical data collected satisfy the requirements of all data users. A summary  
24    of the Quality Control Samples and the associated acceptance criteria is included in Table B3-3.  
25    Key data-quality indicators for laboratory measurements are defined below.

26    Precision

27    Precision shall be assessed by analyzing laboratory duplicates and replicate analyses of  
28    laboratory-control samples and PDP blind-audit samples. Results from measurements on these  
29    samples must be compared to the criteria listed in Table B3-2. These QC measurements will be  
30    used to demonstrate acceptable method performance and to trigger corrective action when  
31    control limits are exceeded.

1     Accuracy

2     Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP blind-audit  
3     samples and laboratory-control samples. Results from these measurements must be compared  
4     to the criteria listed in Table B3-2. These QC measurements will be used to demonstrate  
5     acceptable method performance and to trigger corrective action when control limits are  
6     exceeded.

7     Calibration

8     GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
9     using the procedures and criteria specified in Table B3-3. These criteria will be used to  
10    demonstrate acceptable calibration and to trigger corrective action when control limits are  
11    exceeded.

12    Method Detection Limit

13    MDLs shall be expressed in nanograms for VOCs and must be less than or equal to those listed  
14    in Table B3-2. MDLs shall be determined based on the method described in Section B3-1. The  
15    detailed procedures for MDL determination shall be included in site SOPs.

16    Program Required Quantitation Limit

17    Laboratories must demonstrate the capability to quantitate analytes at or below the PRQLs  
18    given in Table B3-2. Laboratories shall set the concentration of at least one calibration standard  
19    below the PRQL. The detailed procedures for PRQL demonstration shall be included in  
20    laboratory SOPs.

21    Completeness

22    Laboratory completeness shall be expressed as the number of samples analyzed with valid  
23    results as a percent of the total number of samples submitted for analysis. A composited sample  
24    is treated as one sample for the purposes of completeness, because only one sample is run  
25    through the analytical instrument. Valid results are defined as results that meet the data  
26    useability criteria based on application of the Quality Control Criteria specified in Tables B3-2  
27    and B3-3; and meet the detection limit, calibration representativeness, and comparability criteria  
28    within this section. The Permittees shall require that participating laboratories meet the  
29    completeness criteria specified in Table B3-2.

30    Comparability

31    For VOC analysis, data generated through analysis of samples from different sites shall be  
32    comparable. The Permittees shall require each site to achieve comparability by using  
33    standardized methods and traceable standards and by requiring all sites to successfully  
34    participate in the PDP (DOE, 2003).

1 Representativeness

2 Representativeness for VOC analysis shall be achieved by collecting sufficient numbers of  
3 samples using clean sampling equipment that does not introduce sample bias. Samples must  
4 be collected as described in Permit Attachment B1.

5 B3-6 Total Volatile Organic Compound Analysis

6 Quality Assurance Objectives

7 The development of DQOs specifically for this program has resulted in the QAOs listed in Table  
8 B3-4. The specified QAOs represent the required quality of data necessary to draw valid  
9 conclusions regarding program objectives. WAP-required limits, such as the PRQL associated  
10 with VOC analysis, are specified to ensure that the analytical data collected satisfy the  
11 requirements of all data users. Key data-quality indicators for laboratory measurements are  
12 defined below.

13 Precision

14 Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates,  
15 replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from  
16 measurements on these samples must be compared to the criteria listed in Table B3-4. These  
17 QC measurements will be used to demonstrate acceptable method performance and to trigger  
18 corrective action when control limits are exceeded.

19 Accuracy

20 Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control  
21 samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from  
22 these measurements for matrix spikes samples must be compared to the %R criteria listed in  
23 Table B3-4. Results for surrogates and internal standards are evaluated as specified in the SW-  
24 846 method (EPA 1996) or Table B3-5. These QC measurements will be used to demonstrate  
25 acceptable method performance and to trigger corrective action when control limits are  
26 exceeded.

27 Laboratory blanks shall be assessed to determine possible laboratory contamination and are  
28 evaluated as specified in Table B3-5. These QC measurements will be used to demonstrate  
29 acceptable levels of laboratory contamination and to trigger corrective action when control limits  
30 are exceeded.

31 Calibration

32 GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated  
33 using the procedures and criteria specified in Table B3-5 and the SW-846 method (EPA 1996).  
34 These criteria will be used to demonstrate acceptable calibration and to trigger corrective action  
35 when control limits are exceeded.

Method Detection Limit

MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less than or equal to those listed in Table B3-4. The detailed procedures for MDL determination shall be included in site SOPs.

Program Required Quantitation Limit

Laboratories must demonstrate the capability to quantitate analytes in samples at or below the PRQLs given in Table B3-4. Laboratories shall set the concentration of at least one calibration standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in laboratory SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Valid results are defined as results that meet the data useability criteria based upon application of the Quality Control Criteria specified in Tables B3-4 and B3-5 and meet the calibration, detection limit, representativeness, and comparability criteria within this section. Participating laboratories must meet the completeness criteria specified in Table B3-4.

Comparability

For VOC analysis, data generated through analysis of samples from different sites shall be comparable. The Permittees shall require sites to achieve comparability by using standardized SW-846 sample preparation and methods that meet the QAO requirements in Tables B3-4 and B3-5, traceable standards, and by requiring all sites to successfully participate in the PDP (DOE, 2005). Generator/storage sites may use the most recent version of SW-846. Any changes to SW-846 methodology that results in the elimination of sample preparation or analytical methods in use at generator/storage sites must be addressed as a corrective action to address the comparability of data before and after the SW-846 modification.

Representativeness

Representativeness for VOC analysis shall be achieved by collecting unbiased samples. Samples must be collected as described in Permit Attachment B1.

B3-7 Total Semivolatile Organic Compound Analysis

Quality Assurance Objectives

The development of DQOs specifically for this program has resulted in the QAOs listed in Table B3-6. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. WAP-required limits, such as the PRQLs, are specified to ensure that the analytical data collected satisfy the requirements of all data users. A summary of Quality Control Samples and associated acceptance criteria for this analysis is

included in Table B3-7. Key data-quality indicators for laboratory measurements are defined below.

### Precision

Precision shall be assessed by analyzing laboratory duplicates or matrix spike duplicates, replicate analyses of laboratory control samples, and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table B3-6. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

### Accuracy

Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from these measurements for matrix spikes samples must be compared to the %R criteria listed in Table B3-6. Results for surrogates and internal standards are evaluated as specified in the SW-846 method (EPA 1996) or Table B3-7. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Laboratory blanks shall be assessed to determine possible laboratory contamination and are evaluated as specified in Table B3-7. These QC measurements will be used to demonstrate acceptable levels of laboratory contamination and to trigger corrective action when control limits are exceeded.

### Calibration

GC/MS Tunes, Initial Calibrations, and Continuing Calibration will be performed and evaluated using the procedures and criteria specified in Table B3-7 and the SW-846 method (EPA 1996). These criteria will be used to demonstrate acceptable calibration and to trigger corrective action when control limits are exceeded.

### Method Detection Limit

MDLs shall be expressed in mg/kg for SVOCs and must be less than or equal to those listed in Table B3-6. The detailed procedures for MDL determination shall be included in site SOPs.

### Program Required Quantitation Limit

Laboratories must demonstrate the capability to quantitate analytes in samples at or below the PRQLs given in Table B3-6. Laboratories shall set the concentration of at least one calibration standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in laboratory SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Valid results are defined as results that meet the data useability criteria based on application of the Quality Control Criteria specified in Tables B3-6 and B3-7 and meet the detection limit, calibration, representativeness, and comparability criteria within this section. The Permittees shall require participating laboratories to meet the level of completeness specified in Table B3-6.

Comparability

For SVOC analysis, data generated through analysis of samples from different sites shall be comparable. The Permittees shall require sites to achieve comparability by using standardized SW-846 sample preparation and methods that meet the QAO requirements in Tables B3-6 and B3-7, traceable standards, and by requiring all sites to successfully participate in the PDP (DOE, 2005). Generator/storage sites may use the most current version of SW-846 if the methods are consistent with QAO requirements. Any changes to SW-846 methodology that results in the elimination of sample preparation or analytical methods in use at generator/storage sites must be addressed as a corrective action to address the comparability of data before and after the SW-846 modification.

Representativeness

Representativeness for SVOC analysis shall be achieved by collecting unbiased samples. Samples must be collected as described in Permit Attachment B1.

B3-8 Total Metal Analysis

Quality Assurance Objectives

The development of DQOs for the program has resulted in the QAOs listed in Table B3-8. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. WAP-required limits, such as the PRQLs associated with metal analysis, are specified to ensure that the analytical data collected satisfy the requirements of all data users. A summary of Quality Control Samples and the associated acceptance criteria for this analysis is provided in Table B3-9. Key data-quality indicators for laboratory measurements are defined below.

Precision

Precision shall be assessed by analyzing laboratory sample duplicates or laboratory matrix spike duplicates, replicate analyses of laboratory-control samples, and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table B3-8. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

## Accuracy

Accuracy shall be assessed through the analysis of laboratory matrix spikes, PDP blind-audit samples, serial dilutions, interference check samples, and laboratory-control samples. Results from these measurements must be compared to the criterion listed in Table B3-8 and B3-9. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Laboratory blanks and calibration blanks shall be assessed to determine possible laboratory contamination and are evaluated as specified in Table B3-9. These QC measurements will be used to demonstrate acceptable levels of laboratory contamination and to trigger corrective action when control limits are exceeded.

## Calibration

Mass Tunes (for ICP MS only), Standards Calibration, Initial Calibration verifications, and Continuing Calibrations will be performed and evaluated using the procedures and criteria specified in Table B3-9 and the SW-846 method (EPA 1996). These criteria will be used to demonstrate acceptable calibration and to trigger corrective action when control limits are exceeded.

## Program Required Detection Limits

PRDLs, expressed in units of micrograms per L ( $\mu\text{g/L}$ ), are the maximum values for instrument detection limits (**IDL**) permissible for program support under the WAP. IDLs must be less than or equal to the PRDL for the method used to quantitate a specific analyte. Any method listed in Table B-5 of the Waste Analysis Plan (Permit Attachment B) may be used if the IDL meets this criteria. For high concentration samples, an exception to the above requirements may be made in cases where the sample concentration exceeds five times the IDL of the instrument being used. In this case, the analyte concentration may be reported even though the IDL may exceed the PRDL. IDLs shall be determined semiannually (i.e., every six months). Detailed procedures for IDL determination shall be included in laboratory SOPs.

## Program Required Quantitation Limit

The Permittees shall require participating laboratories to demonstrate the capability of analyte quantitation at or below the PRQLs in units of mg/kg wet weight (given in Table B3-8). The PRDLs are set an order of magnitude less than the PRQLs (assuming 100 percent solid sample diluted by a factor of 100 during preparation). The Permittees shall require participating laboratories to set the concentration of at least one QC or calibration standard at or below the solution concentration equivalent of the PRQL. Detailed calibration procedures shall be included in site SOPs.

## Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Valid results are defined as results that meet the data useability criteria based upon application of the Quality

Control Criteria specified in Tables B3-8 and B3-9 and meet the detection limit, calibration, representativeness, and comparability criteria within this section. The Permittees shall require participating laboratories to meet the completeness specified in Table B3-8.

#### Comparability

For metals analysis, data generated through analysis of samples from different sites shall be comparable. Comparability will be achieved by using standardized SW-846 sample preparation and methods that meet QAO requirements in Tables B3-8 and B3-9, demonstrating successful participation in the PDP (DOE, 2005), and use of traceable standards. Generator/storage sites may use the most recent SW-846 update. Any changes to SW-846 methodology that results in the elimination of sample preparation or analytical methods in use at generator/storage sites must be addressed as a corrective action to address the comparability of data before and after the SW-846 modification.

#### Representativeness

Representativeness for metals analysis shall be achieved by the collection of unbiased samples and the preparation of samples in the laboratory using representative and unbiased methods. Samples must be collected as described in Permit Attachment B1.

#### B3-9 Acceptable Knowledge

Acceptable knowledge documentation provides primarily qualitative information that cannot be assessed according to specific data quality goals that are used for analytical techniques. QAOs for analytical results are described in terms of precision, accuracy, completeness, comparability, and representativeness. Appropriate analytical and testing results may be used to augment the characterization of wastes based on acceptable knowledge. To ensure that the acceptable knowledge process is consistently applied, the Permittees shall require sites to comply with the following data quality requirements for acceptable knowledge documentation:

- Precision - Precision is the agreement among a set of replicate measurements without assumption of the knowledge of a true value. The qualitative determinations, such as compiling and assessing acceptable knowledge documentation, do not lend themselves to statistical evaluations of precision. However, the acceptable knowledge information will be addressed by the independent review of acceptable knowledge information during internal and external audits.
- Accuracy - Accuracy is the degree of agreement between an observed sample result and the true value. The percentage of waste containers which require reassignment to a new waste matrix code and/or designation of different hazardous waste numbers based on sampling and analysis data and discrepancies identified by the Permittees during waste confirmation will be reported as a measure of acceptable knowledge accuracy.
- Completeness - Completeness is an assessment of the number of waste streams or number of samples collected to the number of samples determined to be

useable through the data validation process. The acceptable knowledge record must contain 100 percent of the required information (Permit Attachment B4-3). The useability of the acceptable knowledge information will be assessed for completeness during audits.

- Comparability - Data are considered comparable when one set of data can be compared to another set of data. Comparability is ensured through sites meeting the training requirements and complying with the minimum standards outlined for procedures that are used to implement the acceptable knowledge process. All sites must assign hazardous waste numbers in accordance with Permit Attachment B4-3b and provide this information regarding its waste to other sites who store or generate a similar waste stream.
- Representativeness - Representativeness expresses the degree to which sample data accurately and precisely represent characteristics of a population. Representativeness is a qualitative parameter that will be satisfied by ensuring that the process of obtaining, evaluating, and documenting acceptable knowledge information is performed in accordance with the minimum standards established in Permit Attachment B4. Sites also must assess and document the limitations of the acceptable knowledge information used to assign hazardous waste numbers (e.g., purpose and scope of information, date of publication, type and extent to which waste parameters are addressed).

The Permittees shall require each generator/storage site to comply with the nonconformance notification and reporting requirements of Section B3-13 if the results of sampling and analysis specified in Permit Attachment B are inconsistent with acceptable knowledge documentation.

The Permittees shall require each site to address quality control by tracking its performance with regard to the use of acceptable knowledge by: 1) assessing the frequency of inconsistencies among information, and 2) documenting acceptable knowledge inconsistencies identified through radiography, visual examination, headspace-gas analyses, and solidified waste analyses. In addition, the acceptable knowledge process and waste stream documentation must be evaluated through internal assessments by generator/storage site quality assurance organizations and assessments by auditors external to the organization (i.e., the Permittees).

#### B3-10 Data Review, Validation, and Verification Requirements

Procedures shall be developed for the review, validation, and verification of data at the data generation level; the validation and verification of data at the project level; and the verification of data at the Permittee level. Data review determines if raw data have been properly collected and ensures raw data are properly reduced. Data validation verifies that the data reported satisfy the requirements of this WAP and is accompanied by signature release. Data verification authenticates that data as presented represent the sampling and analysis activities as performed and have been subject to the appropriate levels of data review. The requirements presented in this section ensure that WAP records furnish documentary evidence of quality.

The Permittees shall require the sites to generate the following Batch Data Reports for data validation, verification, and quality assurance activities:

- A Testing Batch Data Report or equivalent includes all data pertaining to radiography or visual examination for up to 20 waste containers without regard to waste matrix. Table B3-11 lists all of the information required in Testing Batch Data Reports (identified with an "X") and other information that is necessary for data validation, but is optional in Testing Batch Data Reports (identified with an "O").
  - A Sampling Batch Data Report or equivalent includes all sample collection data pertaining to a group of no more than 20 headspace gas or homogeneous waste samples that were collected for chemical analysis. Table B3-12 lists all of the information required in Sampling Batch Data Reports (identified with an "X") and other information that is necessary for data validation, but is optional in Sampling Batch Data Reports (identified with an "O").
  - An Analytical Batch Data Report or equivalent includes analytical data from the analysis of TRU-mixed waste for up to 20 headspace gas or homogeneous waste samples. Analytical Batch Data Reports or equivalent that contain results for composited headspace gas samples must contain sufficient information to identify the containers that were composited for each composite sample and the sample volume that was taken from each waste container. Because Analytical Batch Data Reports are generated based on the number of samples analyzed, an Analytical Batch Data Report may contain results that are applicable to more than 20 containers depending on how many composite samples are part of the report, but may not exceed a total of 20 samples analyzed. Table B3-13 lists all of the information required in Analytical Batch Data Reports (identified with an "X") and other information that is necessary for data validation, but is optional in Analytical Batch Data Reports (identified with an "O").
- Raw analytical data need not be included in Analytical Batch Data Reports, but must be maintained in the site project files and be readily available for review upon request. Raw data may include all analytical bench sheet and instrumentation readouts for all calibration standard results, sample data, QC samples, sample preparation conditions and logs, sample run logs, and all re-extraction, re-analysis, or dilution information pertaining to the individual samples. Raw data may also include calculation records and any qualitative or semi-quantitative data collected for a sample and that has been recorded on a bench sheet or in a log book.
- An On-line Batch Data Report or equivalent contains the combined information from the Sampling Batch Data Report and Analytical Batch Data Report that is relevant to the on-line method used.

**B3-10a Data Generation Level**

The following are minimum requirements for raw data collection and management which the Permittees shall require for each site:

- All raw data shall be signed and dated in reproducible ink by the person generating it. Alternately, unalterable electronic signatures may be used.
- All data must be recorded clearly, legibly, and accurately in field and laboratory records (bench sheets, logbooks), and include applicable sample identification numbers (for sampling and analytical labs).
- All changes to original data must be lined out, initialed, and dated by the individual making the change. A justification for changing the original data may also be included. Original data must not be obliterated or otherwise disfigured so as not to be readable. Data changes shall only be made by the individual who originally collected the data or an individual authorized to change the data.
- All data must be transferred and reduced from field and laboratory records completely and accurately.
- All field and laboratory records must be maintained as specified in Table B-6 of Attachment B.
- Data must be organized into a standard format for reporting purposes (Batch Data Report), as outlined in specific sampling and analytical procedures.
- All electronic and video data must be stored appropriately to ensure that waste container, sample, and associated QC data are readily retrievable. In the case of classified information, additional security provisions may apply that could restrict retrievability. The additional security provisions will be documented in generator/storage site procedures as outlined in the QAPjP in accordance with prevailing classified information security standards.

Data review, validation, and verification at this level involves scrutiny and signature release from qualified independent technical reviewer(s)<sup>1</sup> as specified below. Individuals conducting this data review, validation, and verification must use checklists that address all of the items included in this section. Checklists must contain or reference tables showing the results of sampling, analytical or on-line batch QC samples, if applicable. Checklists must reflect review of all QC samples and quality assurance objective categories in accordance with criteria established in Tables B3-2 through B3-9 (as applicable to the methods validated). Completed checklists must be forwarded with Batch Data Reports to the project level. Analytical raw data must be available and reviewed by the data generation level reviewer.

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<sup>1</sup>Independent technical review is performed by a competent individual who is not directly responsible for performing the work.

B3-10a(1) Independent Technical Review

The independent technical review ensures by review of raw data that data generation and reduction are technically correct; calculations are verified correct; deviations are documented; and QA/QC results are complete, documented correctly, and compared against WAP criteria. This review validates and verifies all of the work documented by the originator.

One hundred percent of the Batch Data Reports must receive an independent technical review. This review shall be performed by an individual other than the data generator who is qualified to have performed the initial work. The independent technical review must be performed as soon as practicably possible in order to determine and correct negative quality trends in the sampling or analytical process. However at a minimum, the independent technical review must be performed before any waste associated with the data reviewed is managed, stored, or disposed at WIPP, unless the data are being obtained from waste sampling and analysis as containers are being retrieved or generated after initial WSPF approval as described in Attachment B2, Section B2-1. The reviewer(s) must release the data as evidenced by signature, and as a consequence ensure the following:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used (procedure with revision). Data were reported in the proper units and correct number of significant figures.
- Calculations have been verified by a valid calculation program, a spot check of verified calculation programs, and/or 100 percent check of all hand calculations. Values that are not verifiable to within rounding or significant difference discrepancies must be rectified prior to completion of independent technical review.
- The data have been reviewed for transcription errors.
- The testing, sampling, or analytical data QA documentation for Batch Data Reports is complete and includes, as applicable, raw data, DAC and equilibrium calculations and times, calculation records, chain-of-custody (**COC**) forms, calibration records (or references to an available calibration package), QC sample results, and copies or originals of gas canister sample tags. Corrective action will be taken to ensure that all Batch Data Reports are complete and include all necessary raw data prior to completion of the independent technical review.
- QC sample results are within established control limits, and if not, the data have been appropriately qualified in accordance with data useability criteria. Data outside of established control limits will be qualified as appropriate, assigned an appropriate qualifier flag, discussed in the case narrative, and included as appropriate in calculations for completeness. QC criteria that were not met are documented.
- Reporting flags (Table B3-14) were assigned correctly.

- Sample holding time and preservation requirements were met, or exceptions documented.
- Radiography tapes have been reviewed (independent observation) on a waste container basis at a minimum of once per testing batch or once per day of operation, whichever is less frequent (Attachment B1, Section B1-3). The radiography tape will be reviewed against the data reported on the radiography form to ensure that the data are correct and complete.
- Field sampling records are complete. Incomplete or incorrect field sampling records will be subject to resubmittal prior to completion of the independent technical review.
- QAOs have been met according to the methods outlined in Sections B3-2 through B3-9.

#### B3-10b Project Level

Data validation and verification at this level involves scrutiny and signature release from the Site Project Manager (or designee). The Permittees shall require each site to meet the following minimum requirements for each waste container. Any nonconformance identified during this process shall be documented on a nonconformance report (Section B3-13).

The Site Project Manager shall ensure that a repeat of the data generation level review, validation, and verification is performed on the data for a minimum of one randomly chosen waste container quarterly (every three months). This exercise will document that the data generation level review, validation, and verification is being performed according to implementing procedures.

#### B3-10b(1) Site Project Manager Review

The Site Project Manager Review is the final validation that all of the data contained in Batch Data Reports from the data generation level are complete and have been properly reviewed as evidenced by signature release and completed checklists.

One hundred percent of the Batch Data Reports must have Site Project Manager signature release. At a minimum, the Site Project Manager signature release must be performed before any waste associated with the data reviewed is managed, stored, or disposed at WIPP, unless the data are being obtained from waste sampling and analysis as containers are being retrieved or generated as described in Permit Attachment B2, Section B2-1. This signature release must ensure the following:

- The validity of the DAC assignment made at the data generation level based upon an assessment of the data collection and evaluation necessary to make the assignment.
- Testing batch QC checks (e.g., replicate scans, measurement system checks) were properly performed. Radiography data are complete and acceptable based

on evidence of videotape review of one waste container per day or once per testing batch, whichever is less frequent, as specified in B1-3.

- Sampling batch QC checks (e.g., equipment blanks, field duplicates, field reference standards) were properly performed, and meet the established QAOs and are within established data useability criteria.
- Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes, matrix spike duplicates, laboratory control samples) were properly performed and meet the established QAOs and are within established data useability criteria.
- On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-line control samples) were properly performed and meet the established QAOs and are within established data useability criteria.
- Proper procedures were followed to ensure representative samples of headspace gas and homogeneous solids and soil/gravel were taken.
- Data generation level independent technical review, validation, and verification have been performed as evidenced by the completed review checklists and appropriate signature releases.
- Batch data review checklists are complete.
- Batch Data Reports are complete and data are properly reported (e.g., data are reported in the correct units, with the correct number of significant figures, and with qualifying flags).
- Verify that data are within established data assessment criteria and meet all applicable QAOs (Sections B3-2 through B3-9).

**B3-10b(2) Prepare Site Project Manager Summary and Data Validation Summary**

To document the project-level validation and verification described above, the Permittees shall require each Site Project Manager (or designee) to prepare a Site Project Manager Summary and a Data Validation Summary. These reports may be combined to eliminate redundancy. The Site Project Manager Summary includes a validation checklist for each Batch Data Report. Checklists for the Site Project Manager Summary must be sufficiently detailed to validate all aspects of a Batch Data Report that affect data quality. The Data Validation Summary provides verification that, on a per waste container or sample basis as evidenced by Batch Data Report reviews, all data have been validated in accordance with the site QAPjP. The Data Validation Summary must identify each Batch Data Report reviewed (including all waste container numbers), describe how the validation was performed and whether or not problems were detected (e.g., nonconformance reports), and include a statement indicating that all data are acceptable. Summaries must include release signatures.

Once the data have received project-level validation and verification or when the Site Project Manager decides the sample no longer needs to be retained, the Site Project Manager must ensure that the laboratory is notified. Samples must be retained by the laboratory until this notification is received. Gas sample canisters may then be released from storage for cleaning, recertification, and subsequent reuse. Sample tags must be removed and retained in the project files before recycling the canisters. If the Site Project Manager requests that samples or canisters be retained for future use (e.g., an experimental holding time study), the same sample identification and COC forms shall be used and cross-referenced to a document which specifies the purpose for sample or canister retention.

#### B3-10b(3) Prepare Waste Stream Characterization Package

In the event the Permittees request detailed information on a waste stream, the Site Project Manager will provide a Waste Stream Characterization Package. The Site Project Manager must ensure that the Waste Stream Characterization Package (Section B3-12b(3)) will support waste characterization determinations.

#### B3-10c Permittee Level

The final level of data verification occurs at the Permittee level and must, at a minimum, consist of reviewing a sample of the Batch Data Reports during audits of generator/storage sites and Permittee approved laboratories to verify completeness. During such audits, the Permittees are responsible for the verification that Batch Data Reports include the following:

- Project-level signature releases
- Listing of all waste containers being presented in the report
- Listing of all testing, sampling, and analytical batch numbers associated with each waste container being reported in the package
- Analytical Batch Data Report case narratives
- Site Project Manager Summary
- Data Validation Summary
- Complete summarized qualitative and quantitative data for all waste containers with data flags and qualifiers.

For each Waste Stream Profile Form (**WSPF**) submitted for approval, the Permittees must verify that each submittal (i.e., WSPF and Characterization Information Summary) is complete and notify the originating site in writing of the WSPF approval. The Permittees will maintain the data as appropriate for use in the regulatory compliance programs. For subsequent shipments made after the initial WSPF approval, the verification will also include WWIS internal limit checks (Attachment B, Section B-5a(1)).

### B3-11 Reconciliation with Data Quality Objectives

Reconciling the results of waste testing and analysis with the DQOs provides a way to ensure that data will be of adequate quality to support the regulatory compliance programs.

Reconciliation with the DQOs will take place at both the project level and the Permittees' level. At the project level, reconciliation will be performed by the Site Project Manager, while at the Permittees' level, reconciliation will be performed as described below.

#### B3-11a Reconciliation at the Project Level

The Permittees shall require each Site Project Manager to ensure that all data generated and used in decision making meet the DQOs provided in Section B-4a(1) of Permit Attachment B. To do so, the Site Project Manager must assess whether data of sufficient type, quality, and quantity have been collected. The Site Project Manager must determine if the variability of the data set is small enough to provide the required confidence in the results. The Site Project Manager must also determine if, based on the desired error rates and confidence levels, a sufficient number of valid data points have been determined (as established by the associated completeness rate for each sampling and analytical process). In addition, the Site Project Manager must document that random sampling of containers was performed for the purposes of waste stream characterization.

For each waste stream characterized, the Permittees shall require each Site Project Manager to determine if sufficient data have been collected to determine the following WAP-required waste parameters, as applicable:

- Waste matrix code
- Waste material parameter weights
- If each waste container of waste contains TRU radioactive waste
- Mean concentrations,  $UCL_{90}$  for the mean concentrations, standard deviations, and the number of samples collected for each VOC in the headspace gas of waste containers in the waste stream
- Mean concentrations,  $UCL_{90}$  for the mean concentrations, standard deviations, and number of samples collected for VOCs, SVOCs, and metals in the waste stream
- Whether the waste stream exhibits a toxicity characteristic (**TC**) under 40 CFR Part 261, Subpart C
- Whether the waste stream contains listed waste found in 20.4.1.200 NMAC incorporating 40 CFR Part 261, Subpart D
- Whether the waste stream can be classified as hazardous or nonhazardous at the 90-percent confidence level

- 1           ●       Whether an appropriate packaging configuration and DAC were applied and  
2                   documented in the headspace gas sampling documentation, and whether the  
3                   drum age was met prior to sampling.
- 4           ●       Whether all TICs were appropriately identified and reported in accordance with  
5                   the requirements of Section B3-1 prior to submittal of a WSPF for a waste stream  
6                   or waste stream lot.
- 7           ●       Whether the overall completeness, comparability, and representativeness QAOs  
8                   were met for each of the analytical and testing procedures as specified in  
9                   Sections B3-2 through B3-9 prior to submittal of a WSPF for a waste stream or  
10                  waste stream lot.
- 11          ●       Whether the PRQLs for all analyses were met prior to submittal of a WSPF for a  
12                  waste stream or waste stream lot.

13       If the Site Project Manager determines that insufficient data have been collected to make the  
14       determinations listed above, additional data collection efforts must be undertaken. The  
15       reconciliation of a waste stream shall be performed, as described in Permit Attachment B4, prior  
16       to submittal of WSPF and Characterization Information Summary to the Permittees for that  
17       waste stream. The Permittees shall not manage, store, or dispose a TRU mixed waste stream  
18       at WIPP unless the Site Project Manager determines that the WAP-required waste parameters  
19       listed above have been met for that waste stream.

20       The statistical procedure presented in Permit Attachment B2 shall be used by participating Site  
21       Project Managers to evaluate and report waste characterization data from the analysis of  
22       homogeneous solids and soil/gravel. The procedure, which calculates  $UCL_{90}$  values, shall be  
23       used to assess compliance with the DQOs in Attachment B, Section B-4a(1) as well as with  
24       RCRA regulations. The procedure must be applied to all laboratory analytical data for total  
25       VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR § 261.24),  
26       data from the analysis of the appropriate metals and organic compounds shall be expressed as  
27       toxicity characteristic leaching procedure (**TCLP**) values or results may also be compared to the  
28       TC levels expressed as total values. These total values will be considered the regulatory  
29       threshold limit (**RTL**) values for the WAP. RTL values are obtained by calculating the  
30       weight/weight concentration (in the solid) of a TC analyte that would give the regulatory  
31       weight/volume concentration (in the TCLP extract), assuming 100-percent analyte dissolution.

#### 32       B3-11b Reconciliation at the Permittee Level

33       The Permittees must also ensure that data of sufficient type, quality, and quantity are collected  
34       to meet WAP DQOs. The Permittees will ensure sufficient data have been collected to  
35       determine if the waste characterization information is adequate to demonstrate the Permittees'  
36       compliance with Attachment B, Section B-4a(1). This is performed during Permittees' review of  
37       the WSPF and Characterization Information Summary.

## B3-12 Data Reporting Requirements

Data reporting requirements define the type of information and the method of transmittal for data transfer from the data generation level to the project level and from the project level to the Permittees.

### B3-12a Data Generation Level

Data shall be transmitted by hard copy or electronically (provided a hard copy is available on demand) from the data generation level to the project level. Transmitted data shall include all Batch Data Reports and data review checklists. The Batch Data Reports and checklists used must contain all of the information required by the testing, sampling, and analytical techniques described in Permit Attachments B1 through B6, as well as the signature releases to document the review, validation, and verification as described in Section B3-10. All Batch Data Reports and checklists shall be in approved formats, as provided in site-specific documentation.

Batch Data Reports shall be forwarded to the Site Project Manager. All Batch Data Reports shall be assigned serial numbers, and each page shall be numbered. The serial number used for Batch Data Reports can be the same as the testing, sampling, or analytical batch number.

QA documentation, including raw data, shall be maintained in either testing, sampling, and analytical facility files, or site project files for those facilities located on site in accordance with the document storage requirements of site approved site QAPjPs. Permittee approved laboratories shall forward testing, sampling, and analytical QA documentation along with Batch Data Reports to the site project office for inclusion in site project files.

### B3-12b Project Level

The site project office shall prepare a WSPF for each waste stream certified for shipment to WIPP based on information obtained from acceptable knowledge and Batch Data Reports, if applicable. In addition, the site project office must ensure that the Characterization Information Summary and the Waste Stream Characterization Package (when requested by the Permittees) are prepared as appropriate. The Site Project Manager must also verify these reports are consistent with information found in analytical batch reports. Summarized testing, sampling, and analytical data are included in the Characterization Information Summary. The contents of the WSPF, Characterization Information Summary, and Waste Stream Characterization Package are discussed in the following sections.

After approval of a WSPF and the associated Characterization Information Summary by the Permittees, the generator/storage site are required to maintain a cross reference of container identification numbers to each Batch Data Report.

A Waste Stream Characterization Package shall be transmitted by hard copy or electronically from the Site Project Manager to the Permittees when requested.

#### B3-12b(1) Waste Stream Profile Form

The Waste Stream Profile Form (WSPF, Figure B-1) shall include the following information:

- Generator/storage site name
- Generator/storage site EPA ID
- Date of audit report approval by NMED (if obtained)
- Original generator of waste stream
- Whether waste is Contact-Handled or Remote-Handled
- The Waste Stream WIPP Identification Number
- Summary Category Group
- Waste Matrix Code Group
- Waste Material Parameter Weight Estimates per unit of waste
- Waste stream name
- A description of the waste stream
- Applicable EPA hazardous waste numbers
- Applicable TRUCON codes
- A listing of acceptable knowledge documentation used to identify the waste stream
- The waste characterization procedures used and the reference and date of the procedure
- Certification signature of Site Project Manager, name, title, and date signed

**B3-12b(2) Characterization Information Summary**

The Characterization Information Summary shall include the following elements, if applicable:

- Data reconciliation with DQOs
- Headspace gas summary data listing the identification numbers of samples used in the statistical reduction, the maximum, mean, standard deviation, UCL<sub>90</sub>, RTL, and associated EPA hazardous waste numbers that must be applied to the waste stream.
- Total metal, VOC, and SVOC analytical results for homogeneous solids and soil/gravel (if applicable).

- TIC listing and evaluation.
- Radiography and visual examination summary to document that all prohibited items are absent in the waste (if applicable).
- A complete listing of all container identification numbers used to generate the WSPF, cross-referenced to each Batch Data Report
- Complete AK summary, including stream name and number, point of generation, waste stream volume (current and projected), generation dates, TRUCON codes, Summary Category Group, Waste Matrix Code(s) and Waste Matrix Code Group, other TWBIR information, waste stream description, areas of operation, generating processes, RCRA determinations, radionuclide information, all references used to generate the AK summary, and any other information required by Permit Attachment B4, Section B4-2b.
- Method for determining Waste Material Parameter Weights per unit of waste.
- List of any AK Sufficiency Determinations requested for the waste stream.
- Certification through acceptable knowledge or testing and/or analysis that any waste assigned the hazardous waste number of U134 (hydrofluoric acid) no longer exhibits the characteristic of corrosivity. This is verified by ensuring that no liquid is present in U134 waste.

#### B3-12b(3) Waste Stream Characterization Package

The Waste Stream Characterization Package includes the following information:

- Waste Stream Profile Form (WSPF, Section B3-12b(1))
- Accompanying Characterization Information Summary (Section B3-12b(2))
- Complete AK summary (Section B3-12b(2))
- Batch Data Reports supporting the characterization of the waste stream and any others requested by the Permittees
- Raw analytical data requested by the Permittees

#### B3-12b(4) WIPP Waste Information System (WWIS) Data Reporting

The WWIS Data Dictionary includes all of the data fields, the field format and the limits associated with the data as established by this WAP. These data will be subjected to edit and limit checks that are performed automatically by the database, as defined in the *WIPP Waste Information System User's Manual for Use by Shippers/Generators* (DOE, 2001). If a container was part of a composite headspace gas sample, the analytical results from the composite

sample must be assigned as the container headspace gas data results, including associated TICs, for every waste container associated with the composite sample.

### B3-13 Nonconformances

The Permittees shall require the status of work and the WAP activities at participating generator/storage sites to be monitored and controlled by the Site Project Manager. This monitoring and control shall include nonconformance identification, documentation, and reporting.

The nonconformances and corrective action processes specified in this section describe procedures between the Permittees and the generator/storage sites.

### Nonconformances

Nonconformances are uncontrolled and unapproved deviations from an approved plan or procedure. Nonconforming items and activities are those that do not meet the WAP requirements, procurement document criteria, or approved work procedures. Nonconforming items shall be identified by marking, tagging, or segregating, and the affected generator/storage site(s) notified. The Permittees shall require participating sites reconcile and correct nonconforming items as appropriate in accordance with the Permittees' Quality Assurance Program Description (**QAPD**). Disposition of nonconforming items shall be identified and documented. The QAPjPs shall identify the person(s) responsible for evaluating and dispositioning nonconforming items and shall include referenced procedures for handling them.

Management at all levels shall foster a "no-fault" attitude to encourage the identification of nonconforming items and processes. Nonconformances may be detected and identified by anyone performing WAP activities, including

- Project staff - during field operations, supervision of subcontractors, data validation and verification, and self-assessment
- Laboratory staff - during the preparation for and performance of laboratory testing; calibration of equipment; QC activities; laboratory data review, validation, and verification; and self-assessment
- QA personnel - during oversight activities or audits

A nonconformance report shall be prepared for each nonconformance identified. Each nonconformance report shall be initiated by the individual(s) identifying the nonconformance. The nonconformance report shall then be processed by knowledgeable and appropriate personnel. For this purpose, a nonconformance report including, or referencing as appropriate, results of laboratory analysis, QC tests, audit reports, internal memoranda, or letters shall be prepared. The nonconformance report must provide the following information:

- Identification of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance

- Method(s) or suggestions for correcting the nonconformance (corrective action)
- Schedule for completing the corrective action
- An indication of the potential ramifications and overall useability the data, if applicable
- Any approval signatures specified in the site nonconformance procedures

The Permittees shall require the Site Project Manager to oversee the nonconformance report process and be responsible for developing a plan to identify and track all nonconformances and report this information to the Permittees. The Site Project Manager is also responsible for notifying project personnel of the nonconformance and verifying completion of the corrective action for nonconformances.

#### Nonconformance to DQOs

For any non-administrative nonconformance related to applicable requirements specified in this WAP which are first identified at the Site Project Manager signature release level (i.e., a failure to meet a data quality objective DQO), the Permittees shall receive written notification within five (5) calendar days of identification and shall also receive a nonconformance report within thirty (30) calendar days of identification of the incident. The Permittees shall require the generator/storage site to implement a corrective action which remedies the nonconformance prior to management, storage, or disposal of the waste at WIPP. The Permittees shall send NMED a monthly summary of nonconformances identified during the previous month, indicating the number of nonconformances received and the generator/storage sites responsible.

#### Permittees' Corrective Action Process

The Permittees shall initiate a corrective action process when internal nonconformances and nonconformances at the generator/storage sites are identified. Activities and processes that do not meet requirements are documented as deficiencies.

When a deficiency is identified by the Permittees, the following process action steps are required:

- The condition is documented on a Corrective Action Report (**CAR**) by the individual identifying the problem.
- The Permittees have designated the CAR Initiator and Assessment Team Leader to review the CAR, determine validity of the finding (determine that a requirement has been violated), classify the significance of the condition, assign a response due date, and issue the CAR to the responsible party.
- The responsible organization reviews the CAR, evaluates the extent and cause of the deficiency and provides a response to the Permittees, indicating remedial actions and actions to preclude recurrence that will be taken.

- 1           ●     The Permittees review the response from the responsible organization and, if  
2           acceptable, communicate the acceptance to the responsible organization.
- 3           ●     The responsible organization completes remedial actions and actions to preclude  
4           recurrence of the condition.
- 5           ●     After all corrective actions have been completed, the Permittees schedule and  
6           perform a verification to ensure that corrective actions have been completed and  
7           are effective. When all actions have been completed and verified as being  
8           effective, the CAR is closed by the CAR Initiator and Assessment Team Leader  
9           on behalf of the Permittees.
- 10          ●     As part of the planning process for subsequent audits and surveillances, past  
11          deficiencies are reviewed and the previous deficient activity or process is subject  
12          to reassessment.

### 13   B3-14 Special Training Requirements and Certifications

14   Before performing activities that affect WAP quality, all personnel are required to receive  
15   indoctrination into the applicable scope, purpose, and objectives of the WAP and the specific  
16   QAOs of the assigned task. Personnel assigned to perform activities for the WAP shall have the  
17   education, experience, and training applicable to the functions associated with the work.  
18   Evidence of personnel proficiency and demonstration of competence in the task(s) assigned  
19   must be demonstrated and documented. All personnel designated to work on specific aspects  
20   of the WAP shall maintain qualification (i.e., training and certification) throughout the duration of  
21   the work as specified in this WAP and applicable QAPjPs/procedures. Job performance shall be  
22   evaluated and documented at periodic intervals, as specified in the implementing procedures.

23   Personnel involved in WAP activities shall receive continuing training to ensure that job  
24   proficiency is maintained. Training includes both education in principles and enhancement of  
25   skills. Each participating site shall include in its QAPjP a description of the procedures for  
26   implementing personnel qualification and training. All training records that specify the scope of  
27   the training, the date of completion, and documentation of job proficiency shall be maintained as  
28   QA Records in the site project file.

29   Analytical laboratory line management must ensure that analytical personnel are qualified to  
30   perform the analytical method(s) for which they are responsible. The minimum qualifications for  
31   certain specified positions for the WAP are summarized in Table B3-10. QAPjPs, or their  
32   implementing SOPs, shall specify the site-specific titles and minimum training and qualification  
33   requirements for personnel performing WAP activities. QAPjPs/procedures shall also contain  
34   the requirements for maintaining records of the qualification, training, and demonstrations of  
35   proficiency by these personnel.

36   An evaluation of personnel qualifications shall include comparing and evaluating the  
37   requirements specified in the job/position description and the skills, training, and experience  
38   included in the current resume of the person. This evaluation also must be performed for  
39   personnel who change positions because of a transfer or promotion as well as personnel  
40   assigned to short-term or temporary work assignments that may affect the quality of the WAP.

QAPJPs/procedures shall identify the responsible person(s) for ensuring that all personnel maintain proficiency in the work performed and identify any additional training that may be required.

#### B3-15 Changes to WAP-Related Plans or Procedures

Controlled changes to WAP-related plans or procedures shall be managed through the document control process described in the QAPD. The Site Project Manager shall review all non-administrative changes and evaluate whether those changes could impact DQOs specified in the Permit. After site certification, any changes to WAP-related plans or procedures that could positively or negatively impact DQOs (i.e., those changes that require prior approval of the Permittees as defined in Attachment B5, Section B5-2) shall be reported to the Permittees within five (5) days of identification by the project level review. The Permittees shall send NMED a monthly summary briefly describing the changes to plans and procedures identified pursuant to this section during the previous month.

#### B3-16 List of References

Currie, Lloyd A. 1968. "Limits for Qualitative Detection and Quantitative Determination." *Analytical Chemistry*, No. 40: pp. 586-93.

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DOE. 2003. Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases. DOE/CAO-95-1076, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

DOE. 2005. Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes. DOE/CBFO-95-1077, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy.

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Fisenne, I. M., et al. 1973. "Least Squares Analysis and Minimum Detection Levels Applied to Multi-Component Alpha Emitting Samples." *Radiochem. Radioanal. Letters*, 16, No. 1: pp. 5-16.

Pasternack B. S. and N. H. Harley. 1971. "Detection Limits for Radionuclides in the Analysis of Multi-Component Gamma-Spectrometric Data." *Nucl. Instr. and Meth*, No. 91: pp. 533-40.

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## **TABLES**

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**TABLE B3-1**  
**WASTE MATERIAL PARAMETERS AND DESCRIPTIONS**

Waste Material Parameter	Description
Iron-based Metals/Alloys	Iron and steel alloys in the waste; does not include the waste container materials
Aluminum-based Metals/Alloys	Aluminum or aluminum-based alloys in the waste materials
Other Metals	All other metals found in the waste materials
Other Inorganic Materials	Nonmetallic inorganic waste including concrete, glass, firebrick, ceramics, sand, and inorganic sorbents
Cellulosics	Materials generally derived from high-polymer plant carbohydrates; (e.g., paper, cardboard, wood, and cloth)
Rubber	Natural or man-made elastic latex materials; (e.g., surgeons' gloves, and leaded rubber gloves)
Plastics (waste materials)	Generally man-made materials, often derived from petroleum feedstock; (e.g., polyethylene and polyvinylchloride)
Organic Matrix	Cemented organic resins, solidified organic liquids and sludges
Inorganic Matrix	Any homogeneous materials consisting of sludge or aqueous-based liquids that are solidified with cement, calcium silicate, or other solidification agents; (e.g., wastewater treatment sludge, cemented aqueous liquids, and inorganic particulates)
Soils/gravel	Generally consists of naturally occurring soils that have been contaminated with inorganic waste materials
Steel (packaging materials)	55-gal (208-L) drums
Plastics (packaging materials)	90-mil polyethylene drum liner and plastic bags

**TABLE B3-2**  
**GAS VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST**  
**AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b,d</sup> (ng)	FTIRS MDL <sup>b</sup> (ppmv)	PRQL (ppmv)	Completeness (%)
Benzene	71-43-2	≤25	70-130	10	5	10	90
Bromoform	75-25-2	≤25	70-130	10	5	10	90
Carbon tetrachloride	56-23-5	≤25	70-130	10	5	10	90
Chlorobenzene	108-90-7	≤25	70-130	10	5	10	90
Chloroform	67-66-3	≤25	70-130	10	5	10	90
1,1-Dichloroethane	75-34-3	≤25	70-130	10	5	10	90
1,2-Dichloroethane	107-06-2	≤25	70-130	10	5	10	90
1,1-Dichloroethylene	75-35-4	≤25	70-130	10	5	10	90
cis-1,2-Dichloroethylene	156-59-2	≤25	70-130	10	5	10	90
trans-1,2-Dichloroethylene	156-60-5	≤25	70-130	10	5	10	90
Ethyl benzene <sup>d</sup>	100-41-4	≤25	70-130	10	10	10	90
Ethyl ether	60-29-7	≤25	70-130	10	5	10	90
Methylene chloride	75-09-2	≤25	70-130	10	5	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤25	70-130	10	5	10	90
Tetrachloroethylene	127-18-4	≤25	70-130	10	5	10	90
Toluene	108-88-3	≤25	70-130	10	5	10	90
1,1,1-Trichloroethane	71-55-6	≤25	70-130	10	5	10	90
Trichloroethylene	79-01-6	≤25	70-130	10	5	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤25	70-130	10	5	10	90
m-Xylene <sup>c</sup>	108-38-3	≤25	70-130	10	5	10	90
o-Xylene	95-47-6	≤25	70-130	10	5	10	90
p-Xylene <sup>c</sup>	106-42-3	≤25	70-130	10	5	10	90
Acetone	67-64-1	≤25	70-130	150	50	100	90
Butanol	71-36-3	≤25	70-130	150	50	100	90
Methanol	67-56-1	≤25	70-130	150	50	100	90
Methyl ethyl ketone	78-93-3	≤25	70-130	150	50	100	90
Methyl isobutyl ketone	108-10-1	≤25	70-130	150	50	100	90

<sup>a</sup> Criteria apply to PRQL concentrations.

<sup>b</sup> Values based on delivering 10 mL to the analytical system.

<sup>c</sup> These xylene isomers cannot be resolved by GC/MS.

<sup>d</sup> The ethyl benzene PRQL for FTIRS is 20 ppm

CAS = Chemical Abstract Service  
 %RSD = Percent relative standard deviation  
 RPD = Relative percent difference  
 %R = Percent recovery  
 MDL = Method detection limit (maximum permissible value), for GC/MS and GC/FID; total number of nanograms delivered to the analytical system per sample (nanograms); for FTIRS based on 1 m sample cell  
 PRQL = Program required quantitation limit (parts per million/volume basis)

**TABLE B3-3  
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
FREQUENCIES FOR  
GAS VOLATILE ORGANIC COMPOUND ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet method QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical batch or on-line batch	RPD $\leq 25^b$	Nonconformance if RPD >25
Laboratory blanks or on-line blanks	Daily prior to sample analysis for GC/MS and GC/FID. Otherwise, daily prior to sample analysis and one (1) per analytical batch or on-line	Analyte amounts $\leq 3 \times$ MDLs for GC/MS and GC/FID; $\leq$ PRQL for FTIRS	Flag Data if analyte amounts > 3 x MDLs for GC/MS and GC/FID; > PRQL for FTIRS
Laboratory control samples or on-line control samples	One (1) per analytical batch or on-line batch	70-130 %R	Nonconformance if %R <70 or >130
GC/MS comparison sample (for FTIRS only)	One (1) per analytical or on-line batch	RPD $\leq 25^b$	Nonconformance if RPD > 25
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan
GC/MS	BFB Tune Every 12 hours	Abundance criteria for key ions are met	Repeat Until Acceptable
GC/MS	Minimum 5-point initial calibration (minimum of 5 standards) Initially and as needed	%RSD of response factor for each target analyte <35	Repeat Until Acceptable
GC/MS	Continuing calibration Every 12 hours	%D for all target analytes $\leq 30$ of initial calibration	Repeat Until Acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
GC/FID	Minimum 3-point initial calibration (minimum 3 standards) Initially and as needed	Correlation coefficient $\geq$ 0.99 or %RSD <20 for each target analyte and the retention time of each target analyte within an acceptance criteria defined in the method	Repeat Until Acceptable
GC/FID	Continuing calibration Every 12 hours	%RSD $\leq$ 15%	Repeat Until Acceptable

<sup>a</sup> Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.

<sup>b</sup> Applies only to concentrations greater than the PRQLs listed in Table B3-2.

MDL = Method Detection Limit  
QAO = Quality Assurance Objective  
PDP = Performance Demonstration Program  
PRQL = Program Required Quantitation Limit  
%R = Percent Recovery  
RPD = Relative Percent Difference  
BFB = 4-Bromofluorobenzene  
%D = Percent difference  
%RSD = Percent relative standard deviation

**TABLE B3-4  
VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b</sup> (mg/kg)	PRQL <sup>b</sup> (mg/kg)	Completeness (%)
Benzene	71-43-2	≤45	37-151	1	10	90
Bromoform	75-25-2	≤47	45-169	1	10	90
Carbon disulfide	75-15-0	≤50	60-150	1	10	90
Carbon tetrachloride	56-23-5	≤30	70-140	1	10	90
Chlorobenzene	108-90-7	≤38	37-160	1	10	90
Chloroform	67-66-3	≤44	51-138	1	10	90
1,4-Dichlorobenzene <sup>c</sup>	106-46-7	≤60	18-190	1	10	90
ortho-Dichlorobenzene <sup>c</sup>	95-50-1	≤60	18-190	1	10	90
1,2-Dichloroethane	107-06-2	≤42	49-155	1	10	90
1,1-Dichloroethylene	75-35-4	≤250	D-234 <sup>d</sup>	1	10	90
trans-1,2-Dichloroethylene	156-60-5	≤50	60-150	1	10	90
Ethyl benzene	100-41-4	≤43	37-162	1	10	90
Methylene chloride	75-09-2	≤50	D-221 <sup>d</sup>	1	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46-157	1	10	90
Tetrachloroethylene	127-18-4	≤29	64-148	1	10	90
Toluene	108-88-3	≤29	47-150	1	10	90
1,1,1-Trichloroethane	71-55-6	≤33	52-162	1	10	90
1,1,2-Trichloroethane	79-00-5	≤38	52-150	1	10	90
Trichloroethylene	79-01-6	≤36	71-157	1	10	90
Trichlorofluoromethane	75-69-4	≤110	17-181	1	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60-150	1	10	90
Vinyl chloride	75-01-4	≤200	D-251 <sup>d</sup>	1	4	90
m-xylene	108-38-3	≤50	60-150	1	10	90
o-xylene	95-47-6	≤50	60-150	1	10	90
p-xylene	106-42-3	≤50	60-150	1	10	90
Acetone	67-64-1	≤50	60-150	10 <sup>e</sup>	100	90
Butanol	71-36-3	≤50	60-150	10 <sup>e</sup>	100	90
Ethyl ether	60-29-7	≤50	60-150	10 <sup>e</sup>	100	90
Formaldehyde <sup>f</sup>	50-00-0	≤50	60-150	10 <sup>e</sup>	100	90
Hydrazine <sup>g</sup>	302-01-2	≤50	60-150	10 <sup>e</sup>	100	90
Isobutanol	78-83-1	≤50	60-150	10 <sup>e</sup>	100	90
Methanol	67-56-1	≤50	60-150	10 <sup>e</sup>	100	90
Methyl ethyl ketone	78-93-3	≤50	60-150	10 <sup>e</sup>	100	90
Pyridine <sup>c</sup>	110-86-1	≤50	60-150	10 <sup>e</sup>	100	90

<sup>a</sup> Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

<sup>b</sup> TCLP MDL and PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

<sup>c</sup> Can also be analyzed as a semi-volatile organic compound. If analyzed as a semi-volatile compound, the QAOs of Table B3-6 apply.

<sup>d</sup> Detected; result must be greater than zero.

<sup>e</sup> Estimate, to be determined.

<sup>f</sup> Required only for homogeneous solids and soil/gravel waste from Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.

<sup>g</sup> Required only for homogeneous solids and soil/gravel waste from Oak Ridge National Laboratory and Savannah River Site, if analysis is required to resolve assignment of EPA hazardous waste numbers.

CAS = Chemical Abstract Service  
%RSD = Percent relative standard deviation  
RPD = Relative percent difference  
%R = Percent recovery  
MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)  
PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for benzene assuming a 0.9 oz (25-gram [g]) sample, 0.1 gal (0.5 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilogram)

**TABLE B3-5**  
**SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND**  
**FREQUENCIES FOR VOLATILE ORGANIC COMPOUND ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-4 QAOs	Repeat until acceptable
Laboratory duplicates <sup>b</sup>	One (1) per analytical batch	Meet Table B3-4 precision QAOs	Nonconformance if RPDs > values in Table B3-4
Laboratory blanks	One (1) per analytical batch	Analyte concentrations $\leq 3 \times$ MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes <sup>b</sup>	One (1) per analytical batch	Meet Table B3-4 accuracy QAOs	Nonconformance if %Rs are outside the range specified in Table B3-4
Matrix spike duplicates	One (1) per analytical batch	Meet Table B3-4 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-4
Laboratory control samples	One (1) per analytical batch	Meet Table B3-4 accuracy QAO's	Nonconformance if %R < 80 or > 120
GC/MS Calibration	BFB Tune every 12 hours  5-pt. Initial Calibration initially, and as needed	Abundance criteria met as per method  Calibrate according to SW-846 Method requirements:  %RSD for CCC $\leq 30$ , %RSD for all other compounds $\leq 15\%$  Average response factor (RRF) used if %RSD $\leq 15$ , use linear regression if %RSD > 15; R or R <sup>2</sup> $\geq 0.990$ if using alternative curve  System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds $\geq 0.01$	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
GC/MS Calibration (continued)	Continuing Calibration every 12 hours	<p><math>\%D \leq 20</math> for CCC;</p> <p>SPCC minimum RRF as per SW-846 Method; RRF for all other compounds <math>\geq 0.01</math></p> <p>RT for internal standard must be <math>\pm 30</math> seconds from last daily calibration, internal standard area count must be <math>&gt;50\%</math> and <math>&lt;200\%</math> of last daily calibration</p>	Repeat until acceptable
GC/FID Calibration	<p>3-pt. Initial Calibration initially and as needed</p> <p>Continuing Calibration every 12 hours</p>	<p>Correlation Coefficient <math>\geq 0.990</math> or <math>\%RSD \leq 20</math> for all analytes</p> <p><math>\%D</math> or <math>\%Drift</math> for all analytes <math>\leq 15</math> of expected values,</p> <p>RT <math>\pm 3</math> standard deviations from initial RT calibration per applicable SW-846 Method</p>	Repeat until acceptable.
Surrogate compounds	Each analytical sample	Average $\%R$ from minimum of 30 samples for a given matrix $\pm 3$ standard deviations	Nonconformance if $\%R < (\text{average } \%R - 3 \text{ standard deviation})$ or $> (\text{average } \%R + 3 \text{ standard deviation})$
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

<sup>a</sup> Corrective Action per Section B3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> May be satisfied using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table B3-4.

MDL = Method detection limit  
 QAO = Quality assurance objective  
 PDP = Performance Demonstration Program  
 $\%R$  = Percent recovery  
 RPD = Relative percent difference

**TABLE B3-6**  
**SEMI-VOLATILE ORGANIC COMPOUND TARGET ANALYTE LIST**  
**AND QUALITY ASSURANCE OBJECTIVES**

Compound	CAS Number	Precision <sup>a</sup> (%RSD or RPD)	Accuracy <sup>a</sup> (%R)	MDL <sup>b</sup> (mg/kg)	PRQL <sup>b</sup> (mg/kg)	Completeness (%)
Cresols	1319-77-3	≤50	25-115	5	40	90
1,4-Dichlorobenzene <sup>bc</sup>	106-46-7	≤86	20-124	5	40	90
ortho-Dichlorobenzene <sup>c</sup>	95-50-1	≤64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	≤119	D-172 <sup>d</sup>	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152 <sup>d</sup>	0.3	2.6	90
Hexachloroethane	67-72-1	≤44	40-113	5	40	90
Nitrobenzene	98-95-3	≤72	35-180	5	40	90
Pentachlorophenol	87-86-5	≤128	14-176	5	40	90
Pyridine <sup>c</sup>	110-86-1	≤50	25-115	5	40	90

CAS = Chemical Abstract Service  
 %RSD = Percent relative standard deviation  
 RPD = Relative percent difference  
 %R = Percent recovery  
 MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)  
 PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for nitrobenzene assuming a 100-gram (g) sample, 0.5 gal (2 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilograms)

<sup>a</sup> Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

<sup>b</sup> TCLP MDL and PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

<sup>c</sup> Can also be analyzed as a volatile organic compound

<sup>d</sup> Detected; result must be greater than zero

**TABLE B3-7**  
**SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND**  
**FREQUENCIES FOR SEMI-VOLATILE ORGANIC COMPOUNDS ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-6 QAOs	Repeat until acceptable
Laboratory duplicates <sup>b</sup>	One (1) per analytical batch	Meet Table B3-6 precision QAOs	Nonconformance if RPDs > values in Table B3-6
Laboratory blanks	One (1) per analytical batch	Analyte concentrations $\leq 3 \times$ MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes	One (1) per analytical batch	Meet Table B3-6 accuracy QAOs	Nonconformance if RPDs > values and %Rs outside range in Table B3-6
GC/MS Calibration	<p>DFTPP Tune every 12 hours</p> <p>5-pt. Initial Calibration initially, and as needed</p> <p>Continuing Calibration every 12 hours</p>	<p>Abundance criteria met as per method</p> <p>Calibrate according to SW-846 Method requirements:</p> <p>%RSD for CCC <math>\leq 30</math>, %RSD for all other compounds <math>\leq 15\%</math> Average response factor (RRF) used if %RSD <math>\leq 15</math>, use linear regression if &gt;15; R or R<sup>2</sup> <math>\geq 0.990</math> if using alternative curve</p> <p>System Performance Check Compound (SPCC) minimum RRF as per SW-846 Method; RRF for all other compounds <math>\geq 0.01</math></p> <p>%D <math>\leq 20</math> for CCC,</p> <p>SPCC minimum RRF as per SW-846 Method; RRF for all other compounds <math>\geq 0.01</math></p> <p>RT for internal standard must be <math>\pm 30</math> seconds from last daily calibration, internal standard area count must be &gt;50% and &lt;200% of last daily calibration</p>	Repeat until acceptable

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
GC/ECD Calibration	5-pt. Calibration initially and as needed  Continuing Calibration every 12 hours	Correlation Coefficient $\geq$ 0.990 or %RSD < 20 for all analytes  %D or %Drift for all analytes $\leq$ 15 of expected values,  RT $\pm$ 3 standard deviations of initial RT calibration per applicable SW-846 Method	Repeat until acceptable
Matrix spike duplicates	One (1) per analytical batch	Meet Table B3-6 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-6
Laboratory control samples	One (1) per analytical batch	Meet Table B3-6 accuracy QAO's	Nonconformance if %R < 80 or > 120
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples from a given matrix $\pm$ 3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

<sup>a</sup> Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria.

Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> May be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table B3-6.

MDL = Method Detection Limit  
QAO = Quality Assurance Objective  
PDP = Performance Demonstration Program  
%R = Percent Recovery  
RPD = Relative Percent Difference

**TABLE B3-8  
METALS TARGET ANALYTE LIST  
AND QUALITY ASSURANCE OBJECTIVES**

Analyte	CAS Number	Precision (%RSD or RPD) <sup>a</sup>	Accuracy (%R) <sup>b</sup>	PRDL <sup>d</sup> (µg/L)	PRQL <sup>c</sup> (mg/kg)	Completeness (%)
Antimony	7440-36-0	≤30	80-120	100	100	90
Arsenic	7440-38-2	≤30	80-120	100	100	90
Barium	7440-39-3	≤30	80-120	2000	2000	90
Beryllium	7440-41-7	≤30	80-120	100	100	90
Cadmium	7440-43-9	≤30	80-120	20	20	90
Chromium	7440-47-3	≤30	80-120	100	100	90
Lead	7439-92-1	≤30	80-120	100	100	90
Mercury	7439-97-6	≤30	80-120	4.0	4.0	90
Nickel	7440-02-0	≤30	80-120	100	100	90
Selenium	7782-49-2	≤30	80-120	20	20	90
Silver	7440-22-4	≤30	80-120	100	100	90
Thallium	7440-28-0	≤30	80-120	100	100	90
Vanadium	7440-62-2	≤30	80-120	100	100	90
Zinc	7440-66-6	≤30	80-120	100	100	90

<sup>a</sup> ≤ 30 percent control limits apply when sample and duplicate concentrations are ≥ 10 x IDL for ICP-AES and AA techniques, and ≥ 100 x IDL for Inductively Coupled Plasma—Mass Spectrometry (ICP-MS) techniques. If less than these limits, the absolute difference between the two values shall be less than or equal to the PRQL.

<sup>b</sup> Applies to laboratory control samples and laboratory matrix spikes. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.

<sup>c</sup> TCLP PRQL values are reported in units of mg/l and limits are reduced by a factor of 20.

<sup>d</sup> PRDL set such that it is a factor of 10 below the PRQL for 100 percent solid samples, assuming a 100x dilution during digestion.

CAS = Chemical Abstract Service  
 %RSD = Percent relative standard deviation  
 RPD = Relative percent difference  
 %R = Percent recovery  
 PRDL = Program required detection limit (i.e., maximum permissible value for IDL) (micrograms per liter)  
 PRQL = Program required quantitation limit (milligrams per kilogram)

**TABLE B3-9  
 SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND  
 FREQUENCIES FOR METALS ANALYSIS**

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table B3-8 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	$\leq 3 \times \text{IDL}$ ( $\leq 5 \times \text{IDL}$ for ICP-MS) <sup>b</sup>	Redigest and reanalyze any samples with analyte concentrations which are $\leq 10 \times$ blank value and $\geq 0.5 \times$ PRQL
Matrix spikes	One (1) per analytical batch	Meet Table B3-8 accuracy QAOs	Nonconformance if %R outside the range specified in Table B3-8
Matrix spike duplicates	One (1) per analytical batch	Meet Table B3-8 accuracy and precision QAOs	Nonconformance if RPDs > values and %Rs outside range specified in Table B3-8
ICP-MS Tune (ICP-MS Only)	Daily	4 Replicate %RSD $\leq 5$ ; mass calibration within 0.9 amu; resolution < 1.0 amu full width at 10% peak height	Nonconformance if %RSD > 5; mass calibration > 0.9 amu; resolution > 1.0 amu
Initial Calibration 1 blank, 1 standard (ICP, ICP-MS) 3 standard, 1 blank (GFAA, FLAA) 5 standard, 1 blank (CVAA, HAA)	Daily	90-110 %R (80-120% for CVAA, GFAA, HAA, FLAA) for initial calibration verification solution. Regression coefficient $\geq 0.995$ for FLAA, CVA, GFAA, MAA	Correct problem and recalibrate; repeat initial calibration
Continuing Calibration	Every 10 samples and beginning and end of run	90-110% for continuing calibration verification solution. (80-120% for CVAA, GFAA, HAA, FLAA)	Correct problem and recalibrate; rerun last 10 samples
Internal Standard Area Verification (ICP-MS)	Every Sample	Meet SW-846 Method 6020 criteria	Nonconformance if not reanalyzed at 5 X dilution until criteria are met

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>
Serial Dilution (ICP, ICP-MS)	One (1) per analytical batch	5 X dilution must be $\leq 10\%$ D of initial value for sample $> 50 \times \text{IDL}$	Flag Data if $> 10\%$ and $> 50 \times \text{IDL}$
Interference Correction Verification (ICP, ICP-MS)	Beginning and end of run or every 12 hours (8 for ICP) whichever is more frequent	80-120% recovery for analytes  Note: Acceptance Criteria and Corrective Action apply only if interferences found in samples at levels greater than ICS A Solution	Correct problem and recalibrate, nonconformance if not corrected
Laboratory Control Samples	One (1) per analytical batch	Table B3-8 accuracy QAOs	Redigest and reanalyze for affected analytes; non conformance if not reanalyzed
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

<sup>a</sup> Corrective action per Section B3-13 when final reported QC samples do not meet the acceptance criteria. Nonconformances do not apply to matrix related exceedances.

<sup>b</sup> Applies only to concentrations greater than the PRQLs listed in Table B3-8.

IDL = Instrument Detection Limit  
PDP = Performance Demonstration Program  
PRQL = Program Required Quantitation Limit  
%R = Percent Recovery  
RPD = Relative Percent Difference

**TABLE B3-10**  
**MINIMUM TRAINING AND QUALIFICATIONS REQUIREMENTS <sup>a</sup>**

Personnel	Requirements <sup>a</sup>
Radiography Operators <sup>c</sup>	Site-specific training based on waste matrix codes and waste material parameters; requalification every 2 years
FTIRS Technical Supervisors <sup>b</sup> FTIRS Operators <sup>c</sup>	Site-specific and on-the-job training based on the site-specific FTIRS system; requalification every 2 years
Gas Chromatography Technical Supervisors <sup>b</sup> Gas Chromatography Operators <sup>c</sup>	B.S. or equivalent experience and 6 months previous applicable experience
Gas Chromatography/Mass Spectrometry Operators <sup>c</sup> Mass Spectrometry Operators <sup>c</sup>	B.S. or equivalent experience and 1 year independent spectral interpretation or demonstrated expertise
Gas Chromatography/Mass Spectrometry Technical Supervisors <sup>b</sup> Mass Spectrometry Technical Supervisors <sup>b</sup> Atomic Absorption Spectroscopy Technical Supervisors <sup>b</sup> Atomic Absorption Spectroscopy Operators <sup>c</sup> Atomic Mass Spectrometry Operators <sup>c</sup> Atomic Emission Spectroscopy Operators <sup>c</sup>	B.S. or equivalent experience and 1 year applicable experience
Atomic Mass Spectrometry Technical Supervisors <sup>b</sup>	B.S. and specialized training in Atomic Mass Spectrometry and 2 years applicable experience
Atomic Emission Spectroscopy Technical Supervisors <sup>b</sup>	B.S. and specialized training in Atomic Emission Spectroscopy and 2 years applicable experience.

<sup>a</sup> Based on requirements contained in *USEPA Contract Laboratory Program Statement of Work for Organics Analysis* (Document Number OLM 01.0) and *Statement of Work for Inorganics Analysis* (Document Number ILM 03.0).

<sup>b</sup> Technical Supervisors are those persons responsible for the overall technical operation and development of a specific laboratory technique. QAPjPs shall include the site-specific title for this position.

<sup>c</sup> Operators are those persons responsible for the actual operation of analytical equipment. QAPjPs shall include the site-specific title for this position.

**TABLE B3-11**  
**TESTING BATCH DATA REPORT CONTENTS**

Required Information	Radiography	Visual Examination	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Waste container number	X	X	
Waste stream name and/or number	O	O	
Waste Matrix Code	X	X	Summary Category Group included in waste matrix code
Implementing procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
Container type	O	O	Drums, Standard Waste Box, Ten Drum Overpack, etc.
Video media reference	X	X	Reference to Video media applicable to each container. For visual examination of newly generated waste, video media not required if two trained operators review the contents of the waste container to ensure correct reporting.
Imaging check	O		
Camera check		O	
Audio check	O	O	
QC documentation	X	X	
Verification that the physical form matches the waste stream description and Waste Matrix Code.	X	X	Summary Category Group included in waste matrix code
Comments	X	X	
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.
Verify absence of prohibited items	X	X	

Required Information	Radiography	Visual Examination	Comment
Operator signature and date of test	X	X	Signatures of both operators required for Visual Verification of Acceptable Knowledge
Data review checklists	X	X	All data review checklists will be identified

LEGEND:

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

**TABLE B3-12**  
**SAMPLING BATCH DATA REPORT CONTENTS**

Required Information	Headspace Gas	Solid Sampling	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Waste stream name and/or number	O	O	
Waste Matrix Code		X	Summary Category Group included in Waste Matrix Code
Procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
Container number	X	X	
Container type	O	O	Drums, Standard Waste Box, Ten Drum Overpack, etc.
Sample matrix and type	X	X	
Analyses requested and laboratory	X	X	
Point of origin for sampling	X	X	Location where sample was taken (e.g., building number, room)
Sample number	X	X	
Sample size	X	X	
Sample location	X	X	Location within container where sample is taken. (For HSG, specify what layer of confinement was sampled. For solids, physical location within container.)
Sample preservation	X	X	
Person collecting sample	X	X	
Person attaching custody seal	O	O	May or may not be the same as the person collecting the sample
Chain of custody record	X	X	Original or copy is allowed
Sampling equipment numbers	X	X	For disposable equipment, a reference to the lot

Required Information	Headspace Gas	Solid Sampling	Comment
Drum age	X		Must include all supporting determinative information, including but not limited to packaging date, equilibrium start time, storage temperature, and sampling date/time. If Scenario 3 is used, the packaging configuration, filter diffusivity, liner presence/absence, and rigid liner vent hole diameter used in determining the DAC must be documented. If Scenario 1 and 2 are used together, the filter diffusivity and rigid liner vent hole diameter used in determining the DAC must be documented. If default values are used for retrievably stored waste, these values must clearly be identified as such.
Cross-reference of sampling equipment numbers with associated cleaning batch numbers	O	X	As applicable to the equipment used for the sampling. For disposable equipment, a reference to the lot and procurement records to support cleanliness is sufficient
Drum age	X		
Equilibration time	X		
Verification of rigid liner venting	X		Only applicable to containers with rigid liners
Verification that sample volume taken is small in comparison to the available volume	X		Must include headspace gas volume when it can be estimated
Scale Calibration		O	
Depth of waste		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.
Calculation of core recovery		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a representative sample has been taken.
Co-located core description		X	For newly generated waste, if a sampling method other than coring is used, this is replaced by documentation that a QC sample has been taken.
Time between coring and subsampling		X	Only applicable to coring.
OVA calibration and reading	O		Only applicable to manifold systems. Must be done in accordance with manufacturer's specifications
Field Records	X	X	Must contain the following as applicable to the sampling method used: Collection problems, Sequence of sampling collection, Inspection of the solids sampling area, Inspection of the solids sampling equipment, Coring tool test, random location of sub-sample, canister pressure, and ambient temperature and pressure.
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.

Required Information	Headspace Gas	Solid Sampling	Comment
Operator Signature and date and time of sampling	X	X	
Data review checklists	X	X	All data review checklists will be identified

LEGEND:

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

**TABLE B3-13**  
**ANALYTICAL BATCH DATA REPORT CONTENTS**

Required Information	Headspace Gas	Solid Sampling	Comment
Batch Data Report Date	X	X	
Batch number	X	X	
Sample numbers	X	X	
QC designation for sample	X	X	
Implementing procedure (specific version used)	X	X	If procedure cited contains more than one method, the method used must also be cited. Can use revision number, date, or other means to track specific version used.
QC sample results	X	X	
Sample data forms	X	X	Form should contain reduced data for target analytes and TICs
Chain of custody	X	X	Original or copy
Gas canister tags	X		Original or copy
Sample preservation	X	X	
Holding time		X	
Cross-reference of field numbers to laboratory sample numbers	X	X	
Date and time analyzed	X	X	
Verification of spectra used for results	O	O	Analyst must qualitatively evaluate the validity of the results based on the spectra, can be implemented as a check box for each sample
TIC evaluation	X	X	
Reporting flags, if any	X	X	Table B3-14 lists applicable flags
Case narrative	X	X	
Reference to or copy of associated NCRs, if any	X	X	Copies of associated NCRs must be available.
Operator signature and analysis date	X	X	
Data review checklists	X	X	All data review checklists will be identified

**LEGEND:**

X - Required in batch data report.

O - Information must be documented and traceable; inclusion in batch data report is optional.

**TABLE B3-14**  
**DATA REPORTING FLAGS**

DATA FLAG	INDICATOR
B	Analyte detected in blank (Organics/ Headspace gases)
B	Analyte blank concentration greater than or equal to 20 percent of sample concentration prior to dilution corrections (Metals)
E	Analyte exceeds calibration curve (Organics/ Headspace gases)
J	Analyte less than PRQL but greater than or equal to MDL (Organics/ Headspace gases)
J	Analyte greater than or equal to IDL but less than 5 times the IDL before dilution correction (Metals)
U	Analyte was not detected and value is reported as the MDL (IDL for Metals)
D	Analyte was quantitated from a secondary dilution, or reduced sample aliquot (Organics/ Headspace gases)
Z	One or more QC samples do not meet acceptance criteria
H	Holding time exceeded

## **FIGURES**

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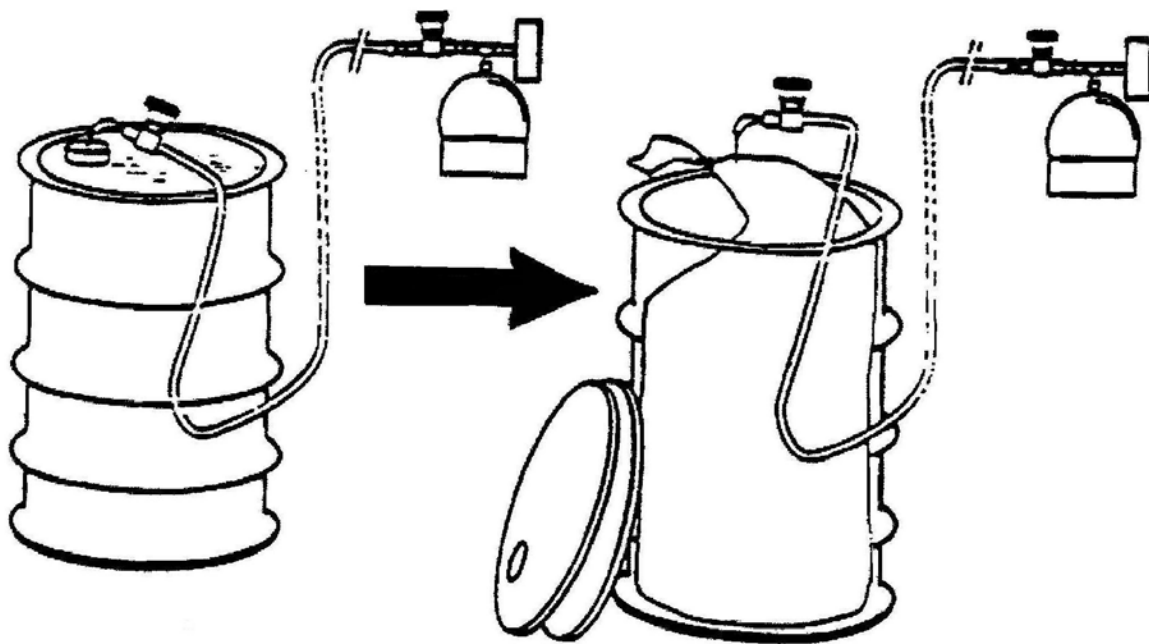


Figure B3-1  
Overall Headspace-Gas Sampling Scheme Illustrating Manifold Sampling